

in which the release coat source and flake deposition source are adapted to alternating vapor deposit layers of the vaporized polymeric release coat material from the release coat source and the vaporized flake material from the flake deposition source on the deposition surface under vacuum to build up in sequence a multi-layer vapor deposit of flake material layers separated by and deposited on corresponding intervening release coat layers;

A 23 the release coat source adapted to apply release coat layers comprising a polymeric material which is vaporized under vacuum to form a smooth continuous solvent soluble and dissolvable barrier layer and support surface on which each of the layers of flake material is formed;

the multi-layer vapor deposit being removable from the vacuum deposition chamber and separating it into flakes by treatment with a solvent which dissolves the release coat layers and yields flakes with smooth, flat surfaces which are essentially free of the release coat material.

REMARKS

The specification has been amended to correct various informalities.

Claims 1 through 18 have been cancelled and new claims 19 through 50 have been added.

Attached hereto is a marked-up version of the changes made to the specification by the current amendment. The attached page is captioned "Version with markings to show changes made."

Respectfully submitted,

CHRISTIE, PARKER & HALE, LLP

By Walter G. Maxwell
Walter G. Maxwell
Reg. No. 25,355
626/795-9900

WGM/agj

VERSION WITH MARKINGS TO SHOW CHANGES MADE

In the Specification:

Please replace the paragraph at page 14, line 15, with the following rewritten paragraph:

The electron beam guns were part of a 15 KV Arco Temiscal 3200 load-lock system. Two mil PET film from SKC was cut into three seventeen inch diameter circles and attached to seventeen inch diameter stainless steel planetary discs located in the vacuum chamber. The chamber was closed and roughed to ten microns then cryopumped to a vacuum of [5X10-7] 5x10-7 Torr.

Please replace the paragraph at page 19, line 14, with the following rewritten paragraph:

Various angstrom scale flake constructions of this invention include (1) aluminum, metal alloy and other metal (described below) monolayer flakes; (2) single layer dielectrics, inorganic or [cross0linked] cross-linked polymer flakes; (3) multi-layer inorganics; (4) optical stacks; (5) inorganic or organic/metal/inorganic or organic multilayer flakes; (6) metal/inorganic/metal flakes/ and (7) CVD or chemically reacted surface coated flakes.

Please replace the paragraph beginning at page 32, line 5 with the following rewritten paragraph:

The following construction was made: A roll of 48 gauge polyester printed with [an] a thermoplastic release coat was metallized in a Temiscal electron beam metallizer with indium. The roll was removed from the metallizer and run through a laboratory stripper using acetone to separate the indium from the polyester. The indium and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics. The flakes in solution were then reduced in particle size using an IKA Ultra Turex T50 Homogenizer. A particle size distribution was taken on the resulting flake using a Horiba LA 910 laser scattering particle size distribution analyzer. The particle [size] sizes reported below are according to the following conventions: D10: 10% of the particles measured are less than or equal to the reported diameter; D50: 50% of the particles measured are less than or equal

to the reported diameter; D90: 90% of the particles measured are less than or equal to the reported diameter. The finished particle size of the flake was D10= 3.3, D50=13.2, D90=31.2.

Please replace the paragraph beginning at page 32, line 29 with the following rewritten paragraph:

The following construction was made: A roll of 48 gauge polyester printed with [an] a thermoplastic release coat was metallized in the Temiscal electron beam metallizer with TiO_2 . The roll was removed from the metallizer and run through a laboratory stripper using acetone to separate the TiO_2 from the polyester. The TiO_2 and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics. The flakes in solution were then reduced in particle size using an IKA Ultra Turex T50 Homogenizer. A particle size distribution was taken on the resulting flake before and after homogenization using a Horiba LA 910 laser scattering particle size distribution analyzer.

Please replace the paragraph beginning at page 33, line 15 with the following rewritten paragraph:

The following construction was made: A roll of 48 gauge polyester printed with [an] a thermoplastic release coat was metallized in the Temiscal electron beam metallizer with MgF_2 . The roll was removed from the metallizer and run through a laboratory stripper using acetone to separate the MgF_2 from the polyester. The MgF_2 and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics. The flakes in solution were then reduced in particle size using an IKA Ultra Turex T50 Homogenizer. A particle size distribution was taken on the resulting flake before and after homogenization using a Horiba LA 910 laser scattering particle size distribution analyzer.

Please replace the paragraph beginning at page 34, line 4 with the following rewritten paragraph:

The following construction was made: A roll of 48 gauge polyester printed with [an] a thermoplastic release coat was metallized in the Temiscal electron beam metallizer with SiO .

The roll was removed from the metallizer and run through a laboratory stripper using acetone to separate the SiO from the polyester. The SiO and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics. The flakes in solution were then reduced in particle size using an IKA Ultra Turex T50 Homogenizer. A particle size distribution was taken on the resulting flake before and after homogenization using a Horiba LA 910 laser scattering particle size distribution analyzer.

Please replace the paragraph beginning at page 34, line 25 with the following rewritten paragraph:

The following construction was made: A roll of 48 gauge polyester printed with [an] a thermoplastic release coat was metallized in the Temiscal electron beam metallizer with ZnO. The roll was removed from the metallizer and run through a laboratory stripper using acetone to separate the ZnO from the polyester. The ZnO and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics. The flakes in solution were then reduced in particle size using an IKA Ultra Turex T50 Homogenizer. A particle size distribution was taken on the resulting flake before and after homogenization using a Horiba LA 910 laser scattering particle size distribution analyzer.

Please replace the paragraph beginning at page 35, line 10 with the following rewritten paragraph:

The following construction was made: A roll of 48 gauge polyester printed with [an] a thermoplastic release coat was metallized in the Temiscal electron beam metallizer with Al₂O₃. The roll was removed from the metallizer and run through a laboratory stripper using acetone to separate the Al₂O₃ from the polyester. The Al₂O₃ and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics. The flakes in solution were then reduced in particle size using an IKA Ultra Turex T50 Homogenizer. A particle size distribution was taken on the resulting flake before and after homogenization using a Horiba LA 910 laser scattering particle size distribution analyzer.

Please replace the paragraph beginning at page 35, line 31 with the following rewritten paragraph:

The following construction was made: A roll of 48 gauge polyester printed with [an] a thermoplastic release coat was metallized in the Temiscal electron beam metallizer with In_2O_3 . The roll was removed from the metallizer and run through a laboratory stripper using acetone to separate the In_2O_3 from the polyester. The In_2O_3 and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics. The flakes in solution were then reduced in particle size using an IKA Ultra Turex T50 Homogenizer. A particle size distribution was taken on the resulting flake before and after homogenization using a Horiba LA 910 laser scattering particle size distribution analyzer.

Please replace the paragraph beginning at page 36, line 19 with the following rewritten paragraph:

The following construction was made: A roll of 48 gauge polyester printed with [an] a thermoplastic release coat was metallized in the Temiscal electron beam metallizer with indium tin oxide (ITO). The roll was removed from the metallizer and run through a laboratory stripper using acetone to separate the ITO from the polyester. The ITO and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics. The flakes in solution were then reduced in particle size using an IKA Ultra Turex T50 Homogenizer. A particle size distribution was taken on the resulting flake before and after homogenization using a Horiba LA 910 laser scattering particle size distribution analyzer.

Please replace the paragraph beginning at page 37, line 3 with the following rewritten paragraph:

The following construction was made: A roll of 48 gauge polyester printed with [an] a thermoplastic release coat was metallized in the Temiscal electron beam metallizer with Si. The roll was removed from the metallizer and run through a laboratory stripper using acetone to

separate the Si from the polyester. The Si and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics. The flakes in solution were then reduced in particle size using an IKA Ultra Turex T50 Homogenizer. A particle size distribution was taken on the resulting flake before and after homogenization using a Horiba LA 910 laser scattering particle size distribution analyzer.

Please replace the paragraph beginning at page 37, line 23 with the following rewritten paragraph:

The following construction was made: A roll of 48 gauge polyester printed with [an] a thermoplastic release coat was metallized in the Temiscal electron beam metallizer with a sandwich of SiO₂/Al/SiO₂. The roll was removed from the metallizer and run through a laboratory stripper using acetone to separate the [Sandwich] Sandwiches from the polyester. The SiO₂/Al/SiO₂ and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics. The flakes in solution were then reduced in particle size using an IKA Ultra Turex T50 Homogenizer. A particle size distribution was taken on the resulting flake before and after homogenization using a Horiba LA 910 laser scattering particle size distribution analyzer.

Please replace the paragraph beginning at page 38, line 6 with the following rewritten paragraph:

The following construction was made: A roll of 48 gauge polyester printed with [an] a thermoplastic release coat was metallized in the Temiscal electron beam metallizer with chromium. The roll was removed from the metallizer and run through a laboratory stripper using acetone to separate the chromium from the polyester. The chromium and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics. The flakes in solution were then reduced in particle size using an IKA Ultra Turex T50 Homogenizer. A particle size distribution was taken on the

resulting flake before and after homogenization using a Horiba LA 910 laser scattering particle size distribution analyzer.

Please replace the paragraph beginning at page 38, line 27 with the following rewritten paragraph:

The following construction was made: A roll of 48 gauge polyester printed with [an] a thermoplastic release coat was metallized in the Temiscal electron beam metallizer with an M-401 copper, zinc, silver alloy, Phelly Materials, Emerson, N.J. The roll was removed from the metallizer and run through a laboratory stripper using acetone to separate the alloy from the polyester. The alloy and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics. The flakes in solution were then reduced in particle size using an IKA Ultra Turex T50 Homogenizer. A particle size distribution was taken on the resulting flake before and after homogenization using a Horiba LA 910 laser scattering particle size distribution analyzer.

Please replace the paragraph beginning at page 40, line 2 with the following rewritten paragraph:

Appendix Example 27

Example: Drum with Polymer Block and E-Beam (wire feed):

Release Material		Styron	Support Material		Aluminum			
Supplier		Dow	Supplier		Mat. Research Corp.			
No.		685D	No.		90101E-AL000-30002			
PVD Conditions:								
E-Beam Power	Release Thickness (Angstroms)	Support Thickness	Drum Speed		Revolutions	Wire Size	Coat Weight	Wire Speed
15%	200	150 Angstroms	1 RPM		100	0.005in./dia	0.0005 grams/in.	6

The following construction was made at the conditions shown above: 48 gauge polyester wrapped around the drum for easy removal was polymer release coated with styrene and metallized in

the Temiscal electron beam metallizer with aluminum. The polyester film was removed from the metallizer and run through a laboratory releasing device using acetone to separate the [Aluminum] aluminum from the releasing layers and the polyester film. The aluminum and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics. The flakes in solution were then reduced in particle size using an IKA Ultra Turex T50 Homogenizer. A particle size distribution was taken on the resulting flake using a Horiba LA 910 laser scattering particle size distribution analyzer.

Please replace the paragraph beginning at page 41, line 2 with the following rewritten paragraph:

Appendix Example 28

Example: Drum with Polymer Block and E-Beam (wire feed):

Release Material		Styron	Support Material		Silicone Dioxide		
Supplier		Dow	Supplier		Cerac		
No.		685D	No.		S-1060		
PVD Conditions:							
E-Beam Power	Release Thickness (Angstroms)	Support Thickness	Drum Speed	Revolutions	Wire Size	Coat Weight	Wire Speed
8%	200	200 Angstroms	1 RPM	100	0.005in. /dia	0.0005 grams/in.	6

The following construction was made at the conditions shown above: 48 gauge polyester wrapped around the drum for easy removal was polymer release coated with styrene and metallized in the Temiscal electron beam metallizer with [Silicone Monoxide] silicone monoxide. The polyester film was removed from the metallizer and run through a laboratory releasing device using acetone to separate the [Silicon Monoxide] silicone monoxide from the releasing layers and the polyester film. The silicon monoxide and acetone solution was then decanted and centrifuged to concentrate the flakes. The resulting flakes were then drawn down on a slide and microphotographed on an Image Pro Plus Image Analyzer from Media Cybernetics.

Please replace the paragraph beginning at page 42 line 34 with the following rewritten paragraph:

FIGS. 15, 16, 15A and 16A show two separate embodiments of a wire feed mechanism for delivering coated polymer to a vacuum chamber which includes a rotating drum, a deposition gauge, a polymer vapor tube with a coated polymer coated wire feed system, and an electron beam (E-beam) gun. The drum is as described previously. The vapor tube is equipped with a heated polymer vapor path surrounded by a water-cooled tube separated by a vacuum gap. A slot in the tubes allows the evaporated polymer to pass through to the drum surface. The vapor tube produces a differential pressure area adjacent the heater block and drum for preventing escape of vapor to the E-beam area of the chamber. In the embodiment shown in FIGS. 15 and 16, the wire feed housing contains a wire supply spool and a take-up spool. The wire is unwound and coated with polymer and runs around the heater block. Polymer is evaporated from the coated wire and is directed onto the drum surface. The end view of FIG. 16 shows the outer tube with its slot facing the drum. The outer tube is cooled and the vapor tube inside is heated. This view also shows the heater block with the wire wrap. The wire passes into the vapor tube, around the heated tube and back out to the take-up spool.

Please replace the paragraph beginning at page 47 line 5 with the following rewritten paragraph:

The goal of the trial was to achieve nanoparticles of aluminum resulting from managing the deposition process such that as the aluminum is deposited on the releasing layer it remains in the [Island] island growth state. These islands of uncoalesced aluminum are then coated with releasing material then recoated with islands of aluminum. This is repeated until a 100 multilayer sandwich of release/aluminum islands/release is formed.

Please replace the paragraph beginning at page 48 line 19 with the following rewritten paragraph:

In one embodiment, the present invention can be used for manufacturing release-coated polymeric carrier film such as release-coated polyester (PET). Referring to FIG. 19, a polyester carrier film 180 is wrapped around a rotating cooling drum 182 contained in a vacuum chamber 184. The film passes from a film unwind station 186 around approximately

300°[C] or more of surface area of the rotating cooling drum, and the coated film is then taken up at a film rewind station 188. A polymer delivery source 190 directs the polymer material toward the carrier film and the E-beam 192 gun vaporizes the polymer for coating it onto the carrier film. The polymeric coating hardens and is then taken up at the rewind station. The process provides a thermoplastic polymeric release-coated heat-resistant polymeric carrier film, in which the film provides good release properties for flake material applied to the film by vapor deposition techniques in a vacuum chamber. The film provides effective release in forming thin flat angstrom level flakes.

Please replace the paragraph beginning at page 52 line 34 to page 53 line 16 with the following rewritten paragraph:

Referring to FIG. 20, the coating apparatus consists of four sections: unwind 200, coating body 202, drying tube 204, and winder 206. The spool of wire is restricted in side to side movement while allowing it to unwind with a minimum of resistance. The coating body comprises a syringe body 208, Becton Dickson 5cc disposable syringe, and a syringe needle 210, Becton Dickson 20GI Precision Glide needle. The disposable syringe is filled with the coating mixture and the needle meters a given amount of material onto the wire. The drying tube is constructed from copper plumbing tubing. From top to bottom the tube consists of a ½ inch tube 212 six inches long, a ½ to ¾ reducer 214, a ¾ inch tube 216 two inches long, a ¾ inch tee 218 from which a 4 inch ¾ inch tube 220 extends perpendicularly. An exhaust fan 222 is attached to this pipe drawing air from the apparatus. The straight section of the tee is attached to a ¾ inch copper tube 224 five feet long. This section is the drying section of the apparatus. Another ¾ inch tee 226 is attached to the 5 foot section. The perpendicular tee is attached to a three inch ¾ inch tube 228 connected to a 90 degree elbow 230 turned upwards. To this elbow is attached a 1 ½ inch tube 232 and a ¾ inch threaded [connector] connector 234. This [connector] connector is attached to a two inch to ¾ inch black iron reducer. A two inch pipe 236 five inches long is screwed into this reducer. The two inch pipe holds the barrel of the hot air gun. The vertical section of the tee is attached to a two inch ¾ inch tube 238, then reduced at 240 to ½ inch. A final six inch section of ½ inch tubing 242 is attached.

Please replace the paragraph beginning at page 53 line 21 with the following rewritten paragraph:

Using the apparatus displayed above the coating is applied to the wire. The wire is unwound from the spool and fed through a syringe body that contains the mixture of polystyrene polymer and solvent. As the wire is [drown] drawn down the syringe body through the syringe needle the wire is covered with the mixture. The coated wire is fed through a copper tube through which heated air is passed. Air is drawn from an exit port in the top of the tube at a rate greater than heated air is supplied from a port in the bottom of the tube. The extra air required by the exhaust port is supplied at the ends of the tube where the wire enters and exits. The amount of hot air supplied to the tube was controlled through the use of a rheostat. It was found 85% of full output was the preferred temperature. Greater temperature caused the coating to blister, less temperature detracted from drying. The wire was wound on a spool after passing through the drying tube. The desired feed rate of the wire was 22 inches per minute through the drying tube. The speed of the winding spool was controlled manually using another rheostat. As more wire was wound onto the spool the rheostat setting was dropped to compensate for the faster pull of the wire during winding. Final coating on the wire was in the 0.4 to 0.5 mg/inch range.

Please replace the paragraph beginning at page 57 line 4 with the following rewritten paragraph:

Based on these data, applications for angstrom scale particles (thickness of less than about 100 [angstrom] angstroms and particles size of less than about 20 microns) for example, may include moisture transmission barrier materials. In use, the flakes line up in parallel in an essentially common plane and produce barriers to water molecules passing through the flake-containing film. Flakes, such as glass flakes, for example, can be used in polymeric films such as PVC, to inhibit plasticizer migration.